Preprint
UCRL-JC-138122

Investigations of Substitutional Impurity Segregation of the ∑5(310)/[001] STGB in FCC Metals: A EFTEM and HRTEM Study

J.M. Plitzko, G.H. Campbell, W.E. King, S.M. Foiles

U.S. Department of Energy

Lawrence Livermore National Laboratory

This article was submitted to 12th European Congress on Electron Microscopy, Brno, Czech Republic, July 9-14, 2000

March 1, 2000

This work was performed under the auspices of the United States Department of Energy by the University of California, Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

This presentation contains no classified nor SUI
Reviewed By: [signature]

Investigations of Substitutional Impurity Segregation to the Σ 5(310)/[001] STGB in FCC Metals: A EFTEM and HRTEM study

Juergen M. Plitzko, Geoffrey H. Campbell, Wayne E. King and Stephen M. Foiles*

Chemistry and Materials Science Directorate, University of California, Lawrence Livermore National Laboratory P.O. Box 808, Livermore, CA 94550

** Computational Materials Science Department, Sandia National Laboratories, MS 9161, Livermore, CA 94551*

Investigations of the Σ 5 symmetric tilt grain boundaries (STGB) in face-centered cubic (FCC) metals in four different metal systems were performed. The metals we have choosen include pure Aluminum, pure Copper, Copper with 1at% Silver, and Aluminum with 1at% Copper. The model grain boundaries have been fabricated with ultra-high vacuum diffusion bonding of single crystals. With the controlled fabrication and preparation of bicrystals we are able to determine composition, structure and morphology of grain boundaries which depends on geometry, crystal orientation, impurity concentration and temperature. The limiting factor in this approach is the ability to fabricate well defined, precisely oriented interfaces, which is enabled here with the UHV Diffusion Bonding Machine [1].

The relation between grain boundary energy and impurity segregation to the interface have been theoretically calculated for the Σ5 (310)/[001] interfaces within the Local Density Approximation (LDA). The calculations use a plane-wave basis and ultrasoft pseudopotentials [2]. The overall structure, especially for the Al interface is qualitatively similar to previous predictions based on pair-potential calculations. These theoretical calculations of the interface structure indicates that the Cu and the Ag atoms segregate to distinct atomic sites at the interface.

High resolution electron microscopy (HRTEM) have been used to reveal the structure of the different interfaces under investigation and to proof the predictions of the theoretical model. Simulations of the HRTEM images were done using these calculated structural models. Additionally structural models based on geometric considerations (coincidence site lattice construction) were used and compared to the experimentally obtained HRTEM images. High resolution images of the boundary structure were acquired using different consecutive defocus values. To acquire these through-focal series we have automated the acquisition process using the script language of the Gatan Digital Micrograph software. The main aim of recording the through-focal series is the possibility of exit wave reconstruction of the investigated boundary structure [3]. Shown in Figure 1a is one of the 30 images of the through-focal series which was obtained with a Philips CM300 FEG UT TEM at the National Center for Electron Microscopy. The homogeneously flat surface of the sample over the field of view and the lack of distortions which are normally present in HRTEM images is a direct result of the sample preparation with a low energy, low angle ion mill [4]. Investigations are planned to compare the reconstructed images (Fig.1b) to 'Z'-contrast images. For these particular investigations we will use the VG HB 603 U in the Solid State Division at the Oak Ridge National Laboratory.

To confirm the presence and determine the amount of the segregant, analytical electron microscopy was performed using a Philips CM 300 FEG ST TEM equipped with an Gatan Imaging Filter (GIF) with a 2kx2k CCD camera and an EDX detector. Electron spectroscopic images were acquired at the $L_{2,3}$-edge of copper around 931 eV to extract elemental maps (three-window-technique) of the copper distribution (Fig. 2a). Because of the small amount of the segregated species and the high energy loss of the investigated ionization edge the resulting elemental distributions are heavily dominated by noise. Series of ESI images were used to enhance the signal and to provide a better background subtraction.

To reveal more precisely the segregation to the grain boundary, EDS line scans were performed and the resulting spectra were quantified (Fig. 2b). The amount of the copper in the analyzed volume of the Al-1at%Cu bicrystal is nearly doubled (≈ 2 at%) in relation to the bulk concentration of ≈ 1 at% (average from different line scans). The quantification results were refined and confirmed by the use of EELS line scans.

This work was performed at Lawrence Livermore National Laboratory under the auspices of the US Dept. of Energy under Contract W-7405-Eng-48

References

1 W. E. King, G. H. Campbell, A. W. Coombs, G. W. Johnson, B. E. Kelly, T. C. Reitz, S. L. Stoner, W. L. Wien and D. M. Wilson in Joining and Adhesion of Advanced Inorganic Materials, edited by A. H. Carim, D. S. Schwartz and R. S. Silberglitt (Mat. Res. Soc. Symp. Proc. 314, Pittsburgh, PA 1993) pp. 61-67.

2 D. Vanderbilt in Physical Review B41, 7892 (1990).

3 A. Thust, W.M.J. Coene, M. Op de Beeck, D. Van Dyck in Ultramicroscopy, 64 211-230 (1996).

4 A. Strecker, J., Mayer, B. Baretzky, U., Eigenthaler, T. Gemming, R. Schweinfest, M. Ruehle in Journal of Electron Micoscopy, 48 [3] 235-244 (1999).

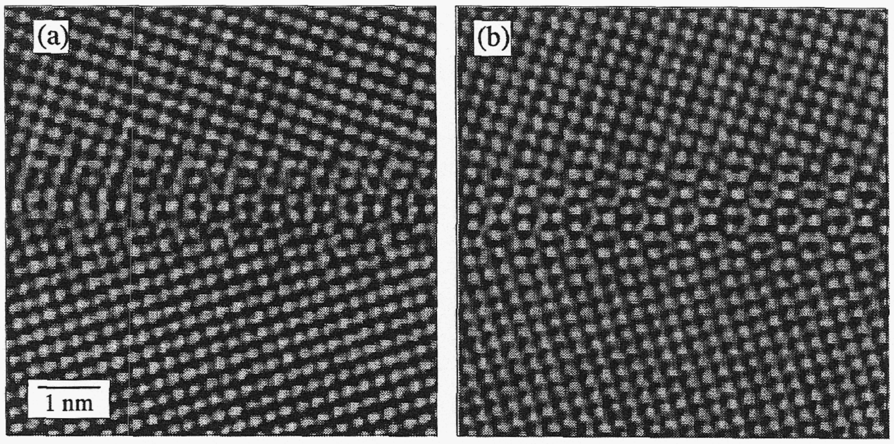

Figure 1: (a) HREM image of $\Sigma 5(310)/[001]$ STGB in Al-1at%Cu. (b) reconstruction of defocus series.

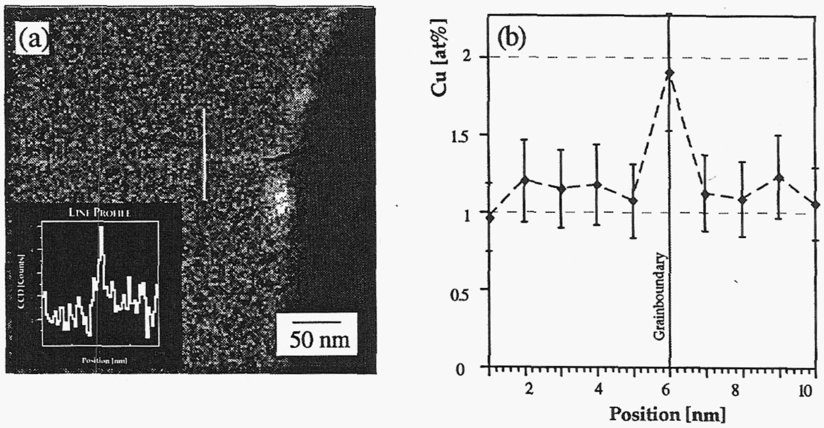

Figure 2: (a) Copper elemental map and (b) illustration of an EDS line scan of Cu concentration across the investigated grain boundary in Al-1at%Cu.